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(54) Method for the production of white sugar of commercial quality from microfiltered or ultrafiltered raw beet juice

(57) Method for the preparation of white sugar of commercial quality from raw beet sugar, including the following operations:

a) microfiltration or ultrafiltration of the juice, after separating the organic and mineral particles whose size is above 50 microns, using membranes whose size is between 5000 MWCO and 0.5 micron;
b) juice sweetening;
c) juice concentration in multi-effect evaporators;
d) cooling crystallisation of the juice thus obtained;
e) separation and washing of the crystals.

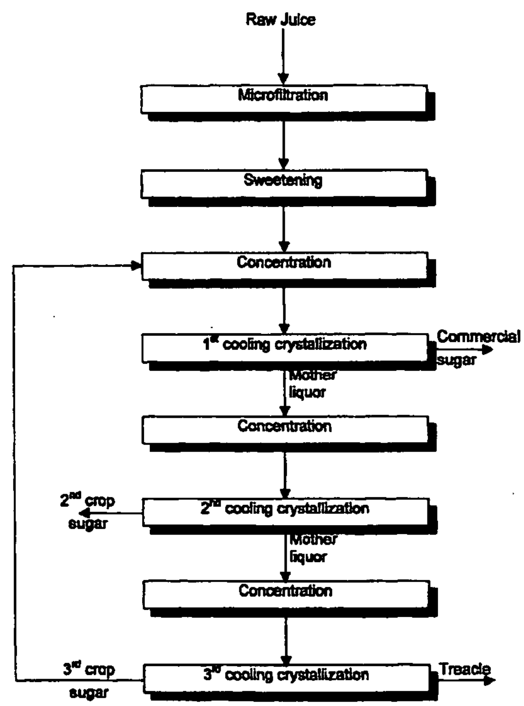

Fig. 1

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Description

[0001] The object of the present invention is an improved method for the production of white sugar of commercial quality from microfiltered or ultrafil-tered raw beet juice.
[0002] According to the traditional working method for beet, the eduction juice (raw juice) undergoes a purging process with lime and carbon dioxide (lime-carbon purging) before being concentrated and going on to the crystallisation stage, the latter being made under evaporation with relatively high temperatures.
[0003] The first sugar coming out of this stage normally undergoes a refining process including sugar dissolution, filtration and, if necessary, juice decolorization and recrystallisation so as to obtain white sugar of commercial quality.
[0004] The lime-carbon purging method involves technologies which have become more and more expensive in recent years because of environmental, plant engineering and energy reasons.
[0005] Other methods have been investigated in order to replace the lime-carbon purging method, but the results obtained for the beet juice purging are not satisfactory at all.
[0006] From USP 5.554.227 a purging method for raw cane or beet sugar is known, in which the juice, after undergoing a clarification treatment, for instance by means of coagulation in order to eliminate colloidal substances, undergoes filtration on diaphragms (microfiltration, ultrafiltration or nanofiltration) and, after sweetening in order to reduce or eliminate the ions Ca^{2+} and Mg^{2+} which are present, said juice is concentrated and then undergoes evaporation crystallisation by means of traditional methods.
[0007] By following the process described above, it is not possible to obtain as a first crop white sugar of commercial quality from beet juice.
[0008] The raw sugar thus obtained has to undergo a refining process including the stages of sugar dissolution, decolorization, filtration, concentration and subsequent recrystallisation.
[0009] According to the present invention it has been found that, by operating under particular condi-tions which will be later better specified, it is possible to obtain a first crop of white sugar of commercial quality from raw beet juice without the necessity of refining stages mentioned above, which is against traditional technique according to which it is not possible to obtain a first crop of white sugar of commercial quality by putting raw beet sugar through microfiltration or ultrafiltration.
[0010] It is known that, in order to obtain white sugar of commercial quality by means of the traditional sugar industry technology, the colour of the starting standard juice should not be much above 500-600 ICUMSA units. Such colour values can be reached:

- by treating with lime-carbon purging the raw sugar obtained from beets which have been produced under particularly favourable climatic conditions;
- by micro(ultra)filtering and subsequent decolorization on absorbing resins of the raw sugar juice;
- presumably, by nanofiltering the raw juice.

[0011] In the crystallisation by evaporation, the sugar crystals thus formed contain considerable amounts of coloured substances and ashes for which subsequent filtration treatments are needed in order to eliminate such substances.
[0012] The patent GB 2206293 describes a method of crystallisation of juices obtained by means of lime-carbon purging, in which the juice is brought under saturation in vacuum at temperatures between 75 and 100°C and then, after adding crystallisation seeds, it is crystallised by means of gradual cooling.
[0013] It is possible to obtain a first crop of white sugar of commercial quality.
[0014] The European patent application no. 96105418.6 describes a method for the preparation of sugar of commercial quality from raw beet juice in which the juice, without being first purged, is directly concentrated within countercurrent evaporators working under vacuum, and the juice thus obtained is crystallised by using the cooling crystallisation tech-nique. Because of the turbidity and of the thermal instability of the juice it is not possible to ob-tain white sugar as first crop; therefore, the sugar has to undergo refining by means of dissolution, filtration and subsequent recrystallisation.
[0015] The juice obtained by micro(ultra)filtration of beet juice is highly coloured (it can reach 5000-6000 and even more ICUMSA units) and contains colour precursor componds which, under the temperature condition used during concentration and crystallisation, can generate coloured compounds. There are also non-sugar compounds which have a negative influence on crystallisation, both slowing it and/or raising sucrose solubility.
[0016] Unexpectedly, it was found that it is possible to obtain white sugar of commercial quality directly through crystallisation of raw beet juice, micro- or ultrafiltered, and then concentrated (after sweetening) in case the crystallisation takes place by means of cooling.
[0017] Moreover, it was found - and this is another unexpected feature of the present invention considering the starting features of the juice - that the three-stage crystallisation of microfiltered juice allows to obtain a highly exhausted final treacle, with a subsequent global crystallisation output which is comparable to the crystallisation output of a traditional

working cycle by means of lime-carbon purging.

[0018] The first sugar which can be obtained by means of the process of the present invention has colour in solution below 40 U.I., which means 53 MEC points and normally below 30 U.I., i.e. 4 MEC points. Representative values are around 27 MEC U.I., i.e. 3,6 MEC points.

Ashes are below 0,1%, i.e. 5,6 MEC points.

Crystal morphology, though being different from that of crystals obtained by means of a tradi-tional working cycle including lime-carbon purging, does not create any problems from the technological point of view.

[0019] The crystals have a slightly elongated shape on axis c) and show a brighter aspect than the crystals with lime-carbon purging.

[0020] The results indicated above are wholly unexpected considering that micro- or ultrafiltered raw beet juice is a highly coloured juice which, ac-cording to the geographical area where the beet is produced, can reach values above 5000-6000 ICUMSA units and which contains a considerable amount of colour precursor compounds forming coloured sub-stances both during the concentration and the crystallisation of the juice. Moreover, the juice contains compounds which are able to delay crystallisation and /or to increase sugar solubility, thus increasing sugar losses in the treacle.

[0021] The method of crystallisation by means of cooling can be carried out in various successive step, each of which includes a concentration and a following crystallisation step.

Figure 1 in the enclosed drawings shows a block diagram of a three-stage crystallisation method according to the present invention.

With reference to said diagram, the method ac-cording to the invention includes a preliminary mi-crofiltration or ultrafiltration stage for the raw sugar, after separation of organic or mineral par-ticles whose size is above 50 micron, and a juice sweetening stage, after which it is possible to go on to the initial stage of the method of the present invention including:

a) the concentration of the juice till saturation is reached with Brix usually between 65 and 75, purity rate between 80 and 90%, working at tem-peratures which are approximately between 70 and 100°C;
b) after reaching the pre-established conditions of hyper-saturation at the temperature at the beginning of crystallisation (example 80°C), it is possible to go on adding to the juice the crystallisation seeds, for instance powdery sucrose suspended in an organic solvent,
c) gradual cooling of the juice, with initially slow cooling speed, faster in the central step and slow again in the final step till a tem-perature of 30-50°C is reached. As a way of example, the temperature gradient is 4/8°C in the first stage, 7-15°C in the second stage and 4-8°C in the third stage.
d) centrifugation and washing of the crystals thus obtained.

[0022] During the concentration stage a) it is preferable to work under vacuum (for example, 0.4 bar abs.)

[0023] The crystals obtained after centrifugation and washing are white sugar of commercial quality. The crystallisation yields depends on the cooling temperature interval and on the Brix value at the beginning of crystallisation.

[0024] The solution deriving from the first crystallisation is concentrated and undergoes a new stage of cooling crystallisation. The profile of the cooling curve is suitably modified, particularly as far as the total time of crystallisation is con-cerned, so as to consider the decreased growth speed of the crystals due to the increased concentration of the non-sugar. Here as well the crystal-lisation yields depends on operative parameters. The second crop sugar, having a particular colour shade and a particular morphology, can be used as a "particular" kind of sugar of commercial quality, it can undergo dissolution and it can be recycled in the concentrated juice and then recrystallised as indicated in the diagram in Fig. 1.

[0025] According to the operative conditions used in the two previous crystallisation stages, to the purity features of the micro- or ultrafiltered juice and from the possible recycling of the second-crop sugar, there can be a third crystallisation stage of the mother liquors deriving from the second crystallisation so as to obtain treacle. Such crystallisation, after concentration, can always be achieved by means of a cooling stage, modifying once more the cooling profile and particularly increasing the total crystallisation time.

[0026] The treacle obtained with three crystallisation stages generally shows a purity rate below 55% with a Brix near 85. The purity rate can reach values considerably below 55% while carrying out the method on an industrial scale.

[0027] Table 1 shows the data relating to the fea-tures of first-, second- and third-crop sugar.

TABLE 1

	First-crop white sugar	Second-crop sugar	Third-crop sugar
Polarisation	99.98	99.68	99.0
Colour in solution (U.I.)	23.2	220.00	757.00
Ashes %	0.0053	0.034	0.073
Inverted %	0.01	0.012	0.015
Farbtype	1.25		

[0028] Table 2 below shows the mass balance.

TABLE 2

Standard at	Mass	S	N	W	Cryst	Brix magma	Sol. rate	Cryst. output	Cryst mother liquor
1st cryst*	100	66.97	7.35	25.68			90.11		
1st cryst	45.04	25.72	7.35	11.96	41.25	86.13	77.78	61.59	0.92
2nd cryst	27.45	14.04	7.35	6.06	11.69	84.52	65.63	45.44	0.43
3rd cryst	17.89	7.91	7.35	2.64	6.13	89.01	51.82	43.47	0.34

[0029] From the mass balance it results that the global crystallisation output can be compared to the output of a traditional working cycle in a sugar plant producing an industrial treacle with rate 60%.
[0030] The distribution of the crystallisation out-puts in the three stages of cooling crystallisation can be varied according to the Brix and temperature conditions at which the crystallisations themselves are regulated.
[0031] In the first crystallisation the output can reach 60% and above.
[0032] As an alternative to the diagram shown in Figure 1, it is possible to use traditional evaporation crystallisation in the production of second- and third-crop sugar, in case such sugars undergo re-working.
As already indicated, the method of the present invention includes the following three successive operations:

- micro- or ultrafiltration of raw juice;
- sugar concentration, after sweetening with cationic resins in order to eliminate or reduce the magnesium and calcium ions;
- crystallisation by cooling of the concentrated juice.

[0033] Before microfiltration the eduction juice of the best is pre-filtered, after being heated at 75-90°C and after pH-stabilisation, in order to eliminate the organic and mineral particles whose size is above 50-100 micron. It may also be settled, with or without using coalescents.
[0034] It may also be possible to add sodium bisulfite, indicatively between 100 and 200 ppm of SO2, both to have a better control on bacterial prolif-eration and to partially neutralise the activity of colour precursor compounds.
[0035] The pre-treated compounds is then microfil-tered or ultrafiltered with a membrane whose pore size is between 5000 MWCO and 0.5 micron, prefera-bly between 20000 MWCO and 0.2 micron.
[0036] The membranes can have a polymeric (both spi-ral and tubular) or an inorganic nature (ceramic membranes).
[0037] The circulation of the flow to be purged takes place beside the wall of the membrane with circula-tion flow rates considerably above those of the permeated compound; this is done so as to minimise dirtying and blocking of the membranes.
[0038] In the preferred "feed and bleed" configuration with various filtration stages, the flow which cannot pass through the membrane pores is fed dur-ing a following stage after the first one, with the possibility, after a certain number of stages, to mix all the possible sucrose during the following stages.
[0039] The operations take place in various stages with temperatures between 75 and 95°C; for instance, operating at 80°C there is no considerable formation of inverted sugar.

[0040] As an example, the membranes which can be used are X-Flow tubular membranes, CELGRAD spiral ultrafiltration membranes, Membralox (U.S.Filter) ceramic diaphragms or CERAM INSIDE from 15000 MWCO to 0.2 microns.

[0041] The permeated compound thus obtained is bacte-riologically sterile. The starting colour is reduced of a considerable value depending on the nature of the juice and on the quality of used membranes. Such decreasing can reach average values of 40-50%, even though there can be considerable fluctuations around this limits.

[0042] The microfiltered juice (it mainly contains Mg^{2+} ions and, in a smaller amount, Ca^{2+} ions) undergoes a sweetening treatment, for instance by passing on strong or weak cationic resins, so as to prevent scaling on the evaporating battery and the precipitation of magnesium oxalates and phosphates.

[0043] The total concentration of Mg^{2+} and Ca^{2+} ions is reduced to values below 5 meq on 100 g of dry product, preferably to 2 meq on 100 g of dry product.

[0044] After sweetening, the juice is concentrated in a multiple effect evaporating battery.

[0045] Because of the thermal instability and the low pH of the micro- or ultrafiltered juice, it may be suitable to carry out the concentration a with countercurrent multiple effect equipment and with the lowest residence times. Even operating with a traditional multiple effect equipment (equicurrent), if the residence times at high temperatures are not particularly high, the juice alterations such as colour increase and sucrose inversion are not such to create problems in obtaining white sugar from the direct cooling crystallisation of the juice.

[0046] The micro- or ultrafiltered juice, then concentrated, thanks to its high bacteriologic purity, can be stocked in tanks as an unfinished product without problems.

[0047] Therefore, it can be directed to crystallisa-tion without having to do that during beet working. Such a method privileges production technologies involving higher residence times which, however, result in simpler and cheaper equipment, with more reliable results.

[0048] In all the operations indicated above the pH value is kept as constant as possible and between 5.5 and 7.5 and preferably between 6.5 and 7.2.

Claims

1. Method for the preparation of white sugar of commercial quality from raw beet sugar, includ-ing the following operations:

   a) microfiltration or ultrafiltration of the juice, after separating the organic and mineral particles whose size is above 50 micron, by means of membranes whose pore size is between 5000 MWCO and 0.5 micron;
   b) juice sweetening;
   c) juice concentration in multiple effect evaporators;
   d) cooling crystallisation of the juice thus obtained;
   e) separation and washing of the crystals.

2. Method according to claim 1, in which the mother juice of the first crystallisation is concentrated till saturation and then undergoes a cooling crystallisation, and the crystals thus ob-tained are redissolved and recycled to the mother juice of the first crystallisation.

3. Method according to claim 2, in which the mother juice of the second crystallisation is concentrated till saturation and then undergoes a cooling crystallisation and the crystals thus obtained are redissolved and recycled to the juice of the first crystallisation.

4. Method according to any of the previous claims 1 to 3, in which the concentration of the juices till saturation is carried out under vacuum.

5. Method according to any of the previous claims 1 to 4, in which the cooling crystallisation is carried out of a gradual cooling, with a temperature gradient of 4-8°C/hour in the first stage, of 7-15°C/hour in the central stage, and again of 4-8°C/hour in the final stage.

6. Method according to the previous claims 1 to 6, in which the juices are concentrated till saturation operating at temperatures between 70 and 100°C and starting from juices with 70-80 Brix.

7. Method according to any of the previous claims 1 to 6, in which an evaporation crystallisation is carried out during the production of first- and second sugar crop.

8. Method according to any of the previous claims 1 to 7, in which the pH value of the juice in the stages from a) to c) is kept at values be-tween 5.5 and 7.5.

9. White sugar obtained according to the method of the previous claims 1 to 8.

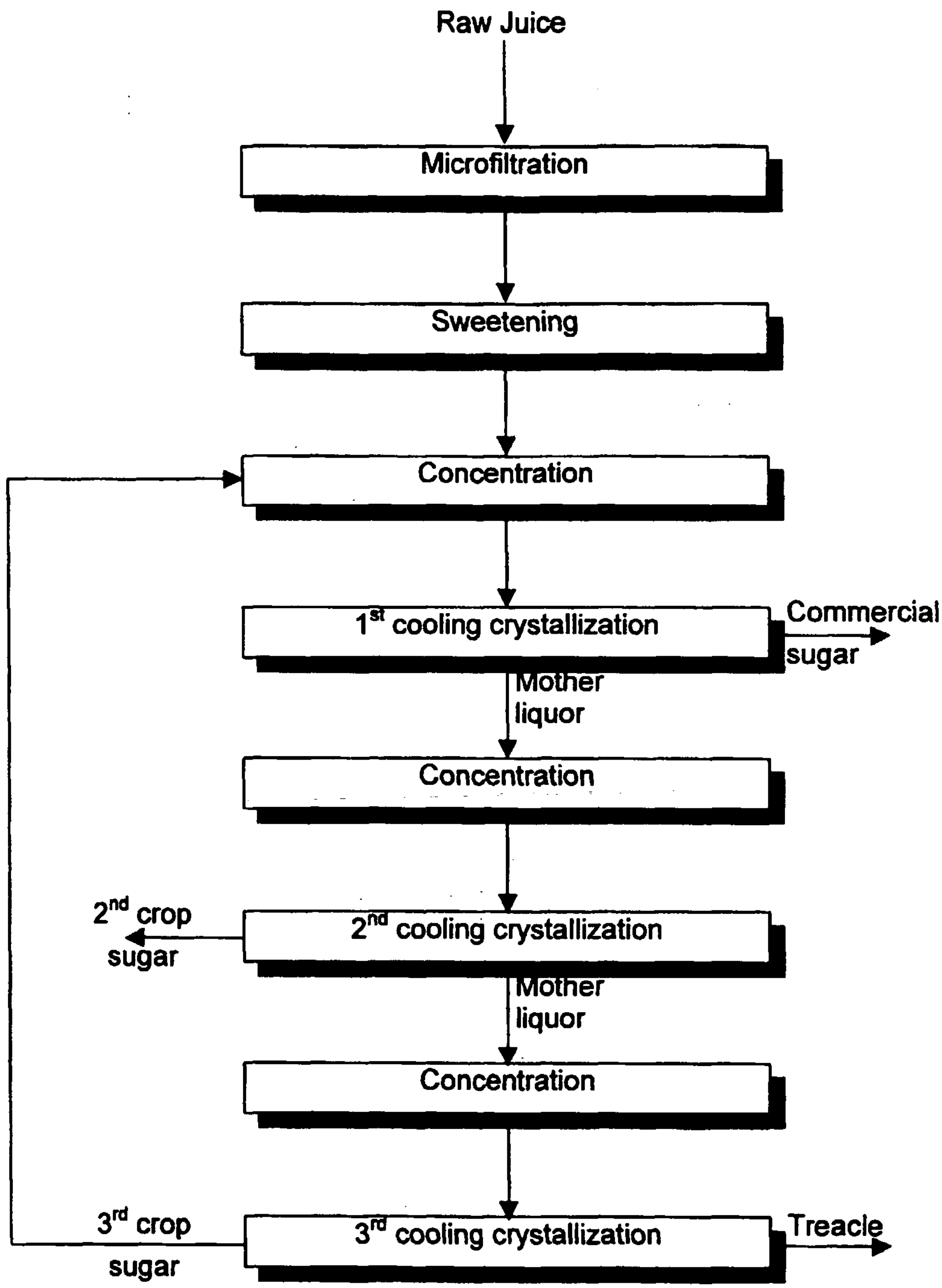
Raw Juice
Microfiltration
Sweetening
Concentration
1st cooling crystallization
Commercial sugar
Mother liquor
Concentration
2nd crop sugar
2nd cooling crystallization
Mother liquor
Concentration
3rd crop sugar
3rd cooling crystallization
Treacle

Fig. 1